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ASD TR 7-772 (VII)

ASD INTERIM REPORT 7-772 (VII)
December 1962

DEVELOPMENT OF MANUFACTURING PROCESS FOR HIGH PURITY ELECTRONIC CERAMICS

J. H. Battle
A. J. Marino, Jr.
E. W. Currier

ITT FEDERAL LABORATORIES DIVISION
International Telephone and Telegraph Corporation

Contract: AF33(600)-42473
ASD Project: 7-772
Interim Technical Progress Report
1 September 1962 - 30 November 1962

The purpose of this project is to develop new or improved manufacturing methods for large scale production of ferroelectric or piezoelectric ceramic materials such as titanates, zirconates, niobates, and tantalates. A major requirement is high purity (99.95%). The accomplishments during this fifth period of Phase II have been the operation of a pilot plant, the chemical and x-ray analyses of its product and the ceramic processing of the powders.

Chemical Engineering Branch
Manufacturing Technology Laboratory
Aeronautical Systems Division
Air Force Systems Command
United States Air Force
Wright-Patterson Air Force Base, Ohio

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ABSTRACT SUMMARY
Interim Technical Progress Report

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DEVELOPMENT OF MANUFACTURING PROCESS FOR HIGH PURITY ELECTRONIC CERAMICS

Physical Sciences Laboratory
ITT Federal Laboratories

The purpose of this project is to develop new or improved manufacturing methods for large scale production of ferroelectric or piezoelectric ceramic materials such as titanates, zirconates, niobates, and tantalates. A major requirement is high purity (99.95%). The accomplishments during this fifth period of Phase II have been the operation of a pilot plant, the chemical and x-ray analyses of its product and the ceramic processing of the powders.

The process for preparing $BaTiO_3$ was frozen, and ten consecutive runs were made. These were analyzed to determine the degree of reproducibility of the process. Results are included.

FOREWORD

This Phase II Interim Report covers the work performed under Contract AF33(600)-42473 from 1 September 1962 to 30 November 1962. It is published for technical information only and does not necessarily represent the recommendations, conclusions or approval of the Air Force.

This contract with the ITT Federal Laboratories, Division of ITT, Nutley, N. J. was initiated under Manufacturing Methods Project 7-772, High Purity Electronic Ceramic Program. It is administered under the direction of Mr. Leo J. Conlon, Project Engineer of the Chemical Engineering Branch, ASRCTC, Manufacturing Technology Laboratory, Aeronautical Systems Division, Wright-Patterson Air Force Base, Ohio.

Mr. P. E. Lighty of the Physical Sciences Laboratory is Project Manager. Others who cooperated in the study and preparation of this report are: Mr. A. J. Marino, Jr., Project Engineer; Mr. J. H. Battle, Chemist; Mr. E. W. Currier, Senior Spectroscopist, Mr. F. E. Pirigyi, Ceramist, and Mr. D. C. Seeley, Chemical Engineer.

The primary objective of this program is to increase the reliability and reproducibility of electronic components fabricated from piezoelectric/ferroelectric materials.

* *

PUBLICATION REVIEW

Reviewed by: _____

P. E. Lighty, Associate Laboratory Director

Approved by: _____

F. A. Muller, Laboratory Director

ASD INTERIM REPORT 7-772 (VII)

DEVELOPMENT OF MANUFACTURING PROCESS FOR

HIGH PURITY ELECTRONIC CERAMICS

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ASD INTERIM REPORT 7-772 (VI)

DEVELOPMENT OF MANUFACTURING PROCESS FOR HIGH PURITY ELECTRONIC CERAMICS

I. INTRODUCTION

This report covers the work done during the interim period 1 September 1962 through 30 November 1962. The information included is the operation of the pilot plant for ten consecutive runs without any changes in the process; chemical analysis; X-ray determinations, and preliminary ceramic data.

II. DISCUSSION

During this period, the process was evaluated by making ten consecutive runs of $BaTiO_3$. Information pertaining to washing times, filter design and volume of wash water was gained. Analysis of all the runs was accomplished by the methods described in previous Interim Reports.[1][2]

A. Pilot Plant Operation - $BaTiO_3$ Synthesis

It was determined from the results of the first twenty runs, that this period would be opportune to "freeze" the process and make ten consecutive batches. The procedure used was described in Interim Report No. V[1], which was a direct scale-up of the laboratory reaction.

Prior to this operation, the reaction kettles were disassembled and inspected for chemical attack and mechanical failures of the linings. There was no noticeable chemical attack, but several areas were cracked. This cracking was caused by stress relief during initial heatup and curing of the resin. The areas affected were the edges of the impellers, valve liners and the valve seats.

As a corrective measure, these areas were cleaned and grooved, where possible, and new resin laid-up. It was given a slow cure before operation. This resulted in a uniform, monolithic, chemical resistant surface, without any of the previous history of small cracks.

The ten runs were carried out according to planned procedure. Two mechanical difficulties occurred causing the abandonment of two runs.

The first problem was a general power failure for twenty minutes during the early part of the digestion period. It was necessary to cool the reaction rapidly to prevent excess alcohol vaporization. The lack of power prevented the stirring of the mass and any possible chance of dropping the reaction into the filter and washing. In order to determine what had happened to the run, it was completed when the power was turned on. The analysis showed it to be off stoichiometry.

A second problem occurred during an additional run where a centrifugal pump failed to transfer all of the barium hydroxide premix into the reactor. This run was aborted due to incomplete reaction.

B. Coprecipitation of $BaSrTiO_3$

In order to show the feasibility of making mixtures of titanates by this direct reaction technique, several bench scale runs were made. The parameters set up during these runs can be used to produce a 80%-20% $BaSrTiO_3$ in the pilot plant.

C. Chemical Analysis

The chemical analysis of barium titanate pilot plant runs continued throughout the period covered by this report. Analyses were made for barium and titanium under the standard methods detailed in previous reports.[1]

Run number 21 was prepared using a lower concentration of potassium hydroxide than normal and the analysis (BaO 64.3%, TiO_2 35.4%) showed a high titanium dioxide content indicating the formation of free TiO_2.

Starting with run number 22 the procedure was frozen at the starting quantities which had been found to give a stoichiometric product in bench scale runs. Chemical analyses for runs 22 through 33 are listed in Table I of this report. Production run number 30 was not analyzed since mechanical failure in the pilot plant required the dumping of the product.

The determination of carbon dioxide in the product was not made during this period. The sensitivity and reproducibility of the procedure outlined in the previous interim report was considered unsatisfactory. Recently, a new type of detector tube was made commercially available, which has been obtained for evaluation. If found satisfactory, it will be used to determine the CO_2 content on all production runs listed in this report.

There was some variation in the potassium content of the products, and, although in all cases it was within specification, experiments were run to see if additional washing would lower the content still further. A 500-gram portion of the barium titanate was washed by suction with 5-1 liter portions of hot (50° to 70° C) water. It was then dried overnight at 100° C and potassium analyses were run by flame photometer. In all cases the potassium content was less than 25 parts per million.

D. Ceramic Processing

A standard procedure for handling $BaTiO_3$ powder was devised. This was done to insure consistency in the analytical sample and the material used for final evaluation.

This procedure is as follows:

1. Pure $BaTiO_3$
   a. Quarter and hand-blend sample
   b. Sieve (-100) 150 to 200 grams of the quartered sample

c. Jet-mill the -100 powder to -325
   (1) 25 to 30 grams of milled powder for analytical examination
   (2) Balance for Ceramic Processing

2. Ceramic Processing of Pure $BaTiO_3$
   a. Add 10% distilled water by weight to the sample. Allow it to stand in a closed container to insure even distribution of moisture.
   b. Mechanically remove lumps before pressing
   c. Weigh out 2 gram samples
   d. Press 1-inch discs at 5,000 psi
   e. Store in plastic boxes or covered containers until fired
   f. Fire as scheduled

3. Ceramic Processing of Mixtures
   To the pure $BaTiO_3$ of 1, C, 2 the following steps are applied.
   a. Weigh out correct ratio of additives for multiples of 100 grams total weight
   b. Ball-mill mixtures:
      (1) Toluene - in a ceramic mill with ceramic balls
      (2) Water - in a rubber mill with rubber balls
   c. Dry powder
   d. Screen through - 100 mesh
   e. Ibid - Procedure 2

Samples were prepared from all pilot plant runs according to sections 1 and 2 of the above procedure. In addition, a number of specimens were prepared omitting the jet-milling step, and others were prepared by ball-milling the -100 mesh $BaTiO_3$ in toluene for one hour. These served as checks on the samples prepared by the standardized procedure.

Composition mixtures were prepared according to procedure 3. The composition chosen for typical body measurements was: 80% $BaTiO_3$, 10% $CaZrO_3$, and 10% $SrTiO_3$.

1. Visual Observation of Fired Discs

In general, the following results were obtained:

a. Samples prepared with jet-milling usually varied in vitrification
b. Samples prepared from jet-milled powders had better vitrification
c. Toluene milling of non-jet-milled powders increased the vitrification
d. Jet-milling and toluene-milling eliminated all inhomogeneities in all samples tested and produced good, dense, vitrified bodies
e. Samples of the stated composition that were jet-milled and toluene-milled were usually more porous than plain $BaTiO_3$. All additives were of high purity.

2. Evaluation of Re-washed Powders

As mentioned, samples from the ten consecutive runs were re-washed to lower further the potassium content. A number of additional pressings and firings of this material were made to study any changes in body characteristics. After re-washing, the powders were jet-milled, pressed, and fired according to schedule. A visual examination of the fired bodies showed them to be more vitrified than the untreated powders.

3. Jet-Mill Changes

With the increased use of the jet-mill for preparing $BaTiO_3$ powder, several modifications were made to improve its performance. They included:

a. The installation of a pre-heater to keep the powder dry on entering the mill
b. A baffle was attached to the feeder to provide a smoother, more uniform feed rate
c. The construction of a larger collection chamber to eliminate excessive back-pressure
d. A high-pressure, high-flow-rate filter and liquid trap was added to the air feed line to prevent any possible contamination from the lines and compressor.

III. CONCLUSION

From the work done in the pilot plant on preparing high purity $BaTiO_3$, it is possible to draw some conclusions about the material.

1. If the process is held constant in all aspects, i.e., raw materials, reaction time, digestion and washing, the product is stoichiometric, uniform in particle size and lattice constants.
2. A combination of wet chemical and X-ray analysis is necessary to achieve precise ratios of $BaO:TiO_2$
3. A high alkaline media is required to produce stoichiometric reactions.
4. Carbon dioxide in the product is found to be adsorbed gas and not chemically bound, which is released at low temperatures.
5. Thickness of filter cake versus volume of water passing through the product is important for the elimination of excess barium and potassium.
6. Firing high purity material is critical. The vitrification temperature is very close to the melting point of the compound.

7. High purity additives such as $CaZrO_3$ tend to increase the vitrification temperature of the plain $BaTiO_3$.

8. Close control of firing is essential to insure dense bodies.

IV BIBLIOGRAPHY

1. ASD Interim Report 7-772(V) June 1962

2. ASD Interim Report 7-772(VI) September 1962

V TABLES

Table I, Impurity Analysis, and Table II, Pilot Plant Data, follow on the next two pages.

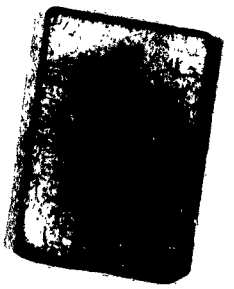

TABLE I

RUN NO.	ASSAY						IMPURITY ANALYSIS											
	CHEMICAL				X-RAY		SPECTROGRAPHIC									FLAME PHOTOMETER		
	BaO_3 TiO_2 ACTUAL		BaO TiO_2 CORRECTED		CORRECTED CHEMICAL		Al_2O_3	CaO	Fe_2O_3	K_2O	MgO	Na_2O	SiO_2	SnO	OTHERS	K	Li	Na
1	61.3	37.0	66.2	33.5	*	*	20	20	10	>1x10⁴	<10	90	10	20	CuO <10	>1x10⁴	–	–
2	62.0	33.5	64.9	35.0	*	*	–	10	10	30	<10	–	10	<10	–	<50	–	–
3	62.2	34.1	64.2	35.2	*	*	–	30	<10	30	<10	30	<10	10	–	>50	–	–
4	62.5	33.5	64.4	34.7	*	*	10	10	10	25	<10	<10	10	<10	–	<50	–	<50
5	66.7	28.6	69.7	30.5	*	*	–	20	10	50	<10	20	10	10	–	<50	–	–
6	63.6	33.0	65.6	34.0	65.5	34.3	10	20	10	25	<10	15	10	10	–	<50	–	<50
7	63.7	32.4	65.8	34.1	*	*	–	20	10	25	<10	15	10	10	–	50	–	–
8	63.4	33.4	65.4	34.5	*	*	10	20	10	50	10	15	20	10	–	400	–	–
9	63.8	32.9	65.8	33.9	*	*	10	20	<10	25	<10	10	<10	10	–	<25	–	–
11	63.4	33.0	65.6	34.1	*	*	<10	20	<10	50	<10	20	<10	10	–	50	–	–
12	55.7	28.1	66.2	33.4	*	*	20	20	20	>300	10	20	10	10	–	>1x10³	–	–
13	63.3	32.1	66.3	33.6	*	*	20	20	10	25	<10	20	10	10	–	80	–	–
14	63.1	32.6	65.8	34.0	*	*	75	20	20	>500	10	20	10	10	–	<1x10³	–	–
15	62.5	33.6	65.0	35.0	65.4	34.5	<100	20	10	25	10	10	10	10	–	35	–	–
17	63.1	34.0	65.4	34.8	65.6	34.8	60	20	10	–	<10	20	10	10	–	100	–	–
18	63.8	33.3	65.4	34.1	65.6	34.8	50	30	<10	25	<10	–	10	20	–	<25	–	–
19	63.8	33.9	65.2	34.6	65.6	34.5	20	20	10	20	<10	10	10	10	–	25	–	–
20	63.5	34.0	65.1	34.8	65.4	34.5	20	20	20	25	<10	10	10	–	–	50	–	–
21	63.1	34.7	64.3	35.7	64.5	35.4	10	30	10	20	<10	20	10	10	–	<25	–	–
22	64.2	33.7	65.6	34.4	65.8	34.3	10	30	10	20	<10	10	10	10	–	<25	–	–
23	64.1	33.5	65.6	34.3	66.0	34.0	10	20	10	40	<10	10	10	10	–	65	–	–
24	64.1	33.1	65.8	34.0	65.9	34.0	10	20	10	60	<10	10	10	10	–	100	–	–
25	64.5	33.0	66.0	33.7	66.3	33.7 (a)	10	20	10	100	<10	30	10	10	–	100	–	–
26	63.9	33.6	65.3	34.4	65.7	34.1	10	20	10	30	<10	10	10	10	–	<25	–	–
27	64.0	33.3	66.0	34.3	66.3	33.7 (a)	10	20	10	30	<10	10	10	10	–	50	–	–
28	63.6	33.4	65.9	34.6	66.1	34.0	10	20	10	80	<10	10	10	10	–	50	–	–
31	64.1	33.0	66.2	34.1	66.4	34.0	40	20	20	30	<10	20	10	–	–	70	–	–
32	63.3	33.3	65.9	34.6	66.1	33.9	10	20	10	70	–	20	10	–	–	100	–	–
33	63.2	33.7	65.5	34.4	66.2	33.9	10	20	10	100	<10	20	10	–	–	200	–	–

a. ANALYTICAL RESULTS AFTER REWASH
25. BaO_3: 65.8 – TiO_2: 34.2
27. BaO_3: 65.7 – TiO_2: 34.2

* X-RAY CORRECTIONS NOT AVAILABLE

TABLE I

PURITY ANALYSIS (PPM)									%		X-RAY		Ignition Loss	REMARKS
		FLAME PHOTOMETER			COLORIMETRIC			GAS EVOLUTION		LATTICE COMP.		@1000°C	
SnO	OTHERS	K	Li	Na	Zn	W	P	CO_2		–	–	%	
20	CuO <10	>1x10⁴	–	–	–	–	–	–		Cubic	$BaTiO_3$	7.5	1ST RUN - HOT TYPICAL
<10	–	<50	–	–	–	–	–	–		Cubic	$BaTiO_3$	4.4	
10	–	>50	–	–	–	–	–	–		Cubic	$BaTiO_3$	3.2	
<10	–	<50	–	<50	–	–	–	–		Cubic	$BaTiO_3$	3.4	
10	–	<50	–	–	–	–	–	–		Cubic	$BaTiO_3$	2.9	
10	–	<50	–	<50	–	–	–	–		Cubic	$BaTiO_3$	3.0	
10	–	50	–	–	–	–	–	–		Cubic	$BaTiO_3$	3.4	
10	–	400	–	–	–	–	–	–		Cubic	$BaTiO_3$	3.1	
10	–	<25	–	–	–	–	–	–		Cubic	$BaTiO_3$	3.0	
10	–	50	–	–	–	–	–	–		Cubic	$BaTiO_3$	3.3	
10	–	>1x10³	–	–	–	–	–	–		Cubic	$BaTiO_3$	15.9	
10	–	80	–	–	–	–	–	–		Cubic	$BaTiO_3$	4.5	
10	–	<1x10³	–	–	–	–	–	–		Cubic	$BaTiO_3$	4.1	
10	–	35	–	–	–	–	–	–		Cubic	$BaTiO_3$	3.9	
10	–	100	–	–	–	–	–	(4) $5x10^{-3}$		Cubic	$BaTiO_3$	2.3	BEGAN N_2 PURGE DURING WASHING
20	–	<25	–	–	–	–	–	(4) $6x10^{-3}$		Cubic	$BaTiO_3$	2.4	SODA LIME SCRUBBERS ON ALL GAS LINES
10	–	25	–	–	–	–	–	(4) $5x10^{-3}$		Cubic	$BaTiO_3$	2.1	
–	–	50	–	–	–	–	–	(4) $2.6x10^{-2}$		Cubic	$BaTiO_3$	2.4	REPAIR TO TANK COATING
10	–	<25	–	–	–	–	–	–		Cubic	$BaTiO_3$	1.9	REVISED HYDROXIDE COATING
10	–	<25	–	–	–	–	–	–		Cubic	$BaTiO_3$	2.1	START OF 10 RUN SERIES
10	–	65	–	–	–	–	–	–		Cubic	$BaTiO_3$	2.3	
10	–	100	–	–	–	–	–	–		Cubic	$BaTiO_3$	2.6	
10	–	100	–	–	–	–	–	–		Cubic	$BaTiO_3$	2.2	
10	–	<25	–	–	–	–	–	–		Cubic	$BaTiO_3$	2.2	
10	–	50	–	–	–	–	–	–		Cubic	$BaTiO_3$	3.0	
10	–	50	–	–	–	–	–	–		Cubic	$BaTiO_3$	2.4	
–	–	70	–	–	–	–	–	–		Cubic	$BaTiO_3$	3.2	
–	–	100	–	–	–	–	–	–		Cubic	$BaTiO_3$	2.5	
–	–	200	–	–	–	–	–	–		Cubic	$BaTiO_3$	2.2	

NOT ENOUGH CAKE

PILOT PLANT DATA

TABLE II

RUN NO	BARIUM CARBONATE lbs	BARIUM HYDROXIDE lbs	H_2O GAL	TITANIUM PRESS. ESTER lbs	TITANIUM PRESS. ALCOH GAL	REACT MIN	DIGESTION MIN	WASH GAL	WASH MIN	WET WGT. LBS	1ST DRY lbs	FINAL WGT. lbs	REMARKS
1	29# 0'	38# 10'	15	62# 9'	15	40	60	40	-	61# 4'	26# 10'	—	1ST RUN - NOT CONSIDERED TYPICAL
2	N/A	N/c	N/c	n/c	10	34	73	126	270	26# 8'	18# 11'	—	TEST RUN FOR STORAGE & FILTRATION
3	N/c	n/c	n/c	n/c	n/c	28	60	90	90	42# 0'	14# 10'	28# 5'	FILTRATION TEST
4	n/c	N/c	n/c	n/c	n/c	30	N/c	185	105	43# 15'	19# 2'	26# 2'	
5	N/c	34# 8'	17	N/c	n/c	28	N/c	65	195	35# 8'	17# 3'	18# 4'	
6	29# 8'	32# 10'	20	63# 11'	N/c	60	N/c	85	85	47# 8'	22# 8'	28# 10'	EXTREMELY FLUID POWDER
7	N/c	N/c	N/c	N/c	n/c	54	N/c	70	120	43# 7'	19# 5'	28# 4'	
8	N/c	n/c	n/c	N/c	n/c	56	N/c	104	149	46# 6'	-	19# 7'	
9	n/c	N/c	n/c	n/c	n/c	70	n/c	n/c	130	51# 15'	25# 9'	34# 11'	
11	n/c	n/c	N/c	n/c	n/c	66	N/c	N/c	139	48# 2'	23# 11'	19# 12'	
12	n/c	n/c	n/c	N/c	N/c	N/c	N/c	100	110	42# 14'	19# 0'	20# 5'	
13	n/c	N/c	N/c	n/c	n/c	n/c	79	60	130	44# 3'	21# 10'	22# 5'	
14	n/c	n/c	n/c	n/c	n/c	N/c	66	40	150	53# 5'	21# 8'	22# 9'	
15	n/c	n/c	n/c	n/c	n/c	n/c	60	70	100	49# 7'	21# 9'	23# 4'	
17	n/c	n/c	n/c	n/c	n/c	63	85	100	117	47# 9'	24# 6'	23# 9'	
18	n/c	n/c	n/c	n/c	n/c	N/c	87	N/c	96	53# 9'	23# 8'	23# 4'	
19	n/c	n/c	n/c	n/c	n/c	65	80	n/c	56	46# 11'	21# 8'	23# 11'	
20	n/c	n/c	n/c	n/c	n/c	66	90	n/c	94	52# 15'	26# 5'	30# 15'	
21	29# 10'	38# 0'	15	61# 15'	n/c	88	62	n/c	105	44# 4'	18# 10'	18# 3'	REDUCED HYDROXIDE CONTENT
22	29# 8'	32# N'	20	63# 11'	N/c	97	70	N/c	125	46# 5'	20# 5'	20# 1'	START OF 10 RUN SERIES
23	N/c	N/c	n/c	n/c	n/c	96	74	90	137	48# 11'	21# 7'	20# 15'	
24	N/c	n/c	n/c	n/c	n/c	95	90	100	87	37# 7'	14# 9'	14# 2'	
25	N/c	n/c	n/c	n/c	n/c	91	150	N/c	122	41# 12'	14# 11'	19# 5'	
26	N/c	n/c	N/c	n/c	n/c	91	88	85	250	53# 2'	26# 4'	24# 11'	CHANGE IN FILTER BAG CONSTRUCTION
27	N/c	N/c	N/c	N/c	N/c	112	84	88	239	53# 10'	27# 1'	24# 2'	
28	N/c	n/c	n/c	n/c	n/c	81	60	90	180	52# 4'	25# 0'	23# 15'	
31	N/c	n/c	n/c	n/c	N/c	n/c	N/c	N/c	195	52# 14'	24# 2'	23# 4'	
32	N/c	n/c	n/c	n/c	n/c	78	N/c	76	288	58# 8'	25# 8'	24# 9'	
33	N/c	N/c	n/c	N/c	N/c	79	n/c	87	185	57# 0'	24# 9'	23# 14'	

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The purpose of this project is to develop new or improved manufacturing methods for large scale production of ferroelectric or piezoelectric ceramic materials such as titanates, zirconates, niobates, and tantalates. A major requirement is high purity (99.95%). The accomplishments during this fifth period of Phase II have been the operation of a pilot plant, the chemical and x-ray analyses of its product and the ceramic processing of the powders.

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1. Chemical Compounds-Barium Compounds
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3. Chemical Reactions-Hydrolysis. Oxalate.
4. Processes and Processing Manufacturing Methods Analytical Methods.
5. Materials- Ceramic Materials
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